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August 26, 2005

NO TSCA CBI

8EHQ-60-373

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Room 6428
Attention: TSCA 8(e) Coordinator
Office of Pollution Prevention and Toxics
U.S. Environmental Protection Agency
1201 Constitution Avenue, N. W.
Washington, D. C. 20460

Contain NO CBI

Re: TSCA Section 8(e) Supplemental Notice: Sulfonate-based and Carboxylic-based Fluorochemicals, Docket Nos. 8EHQ-1180-374; 8EHQ-0381-0394; 8EHQ-0598-373

Dear Sir:

3M is submitting this notice to supplement its previous submissions on sulfonyl and carboxylic-based fluorochemicals.

3M has recently received the enclosed analytical report which presents data from 36 lots of commercially available normal pooled human serum that were purchased by the 3M Environmental Laboratory and subsequently screened for endogenous fluorochemicals. Each sample was analyzed using a protein precipitation technique and screened for sixteen different fluorochemicals, including perfluorooctane sulfonate (PFOS) ($C_8F_{17}SO_3^-$) and perfluorooctanoic acid (PFOA) ($C_7F_{15}COOH$), using liquid chromatography/tandem mass spectrometry (LC/MS/MS). Much of the data presented in the report was derived from extrapolated curves and should be considered as screening quality data only and not necessarily representative of the general population levels. Not all of the detected chemicals are or were produced by 3M. In addition, the levels of fluorochemicals reported are generally corroborative of previously reported values and/or do not represent "substantial risk" information

If you have any questions, please do not hesitate to contact Dale L. Bacon at (651) 778-4736.

Sincerely,

Katherine E Reed TKF

Katherine D. Reed, PhD
Staff Vice President
Environmental Technology and Safety Services

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3M

NO TSCA CBI

Analytical Report

Revised August 12, 2005

Initial Quantitative Screening of Commercial Lots of Human Serum Obtained in 2004 for Endogenous Fluorochemicals Using Protein Precipitation and Liquid Chromatography/Tandem Mass Spectrometry

Laboratory Request Number: E05-0120

Testing Laboratory

3M Environmental Health and Safety Operations
3M Environmental Laboratory

3M Environmental Laboratory

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3M Environmental Laboratory

Analytical Report

Project Number: E05-0120

The following analytical report presents data from commercially available normal pooled human serum that were purchased by the 3M Environmental Laboratory and subsequently screened for endogenous fluorochemicals.

Thirty-six separate lots of pooled normal human serum were extracted using a protein precipitation technique and screened for sixteen different fluorochemicals using liquid chromatography/tandem mass spectrometry (LC/MS/MS). Serum samples were received from various suppliers between 9/9/04 and 12/2/04. Samples were analyzed between 02/03/05 and 02/09/05.

This range-finding study was designed to provide lower limits of quantitation (LLOQs) of approximately 100 - 300 ppt. Upon review of the data, it was discovered that many of the target analytes were present at concentrations much lower than the 100 - 300 ppt target level. As an interim solution and until a more definitive study can be completed, signal to noise ratios were evaluated and a limit of detection (LOD) was defined for each analyte. As a result, calibration curves were extrapolated between the LLOQ and the newly defined LOD, allowing screening quality data to be determined for some analytes as low as 19.9 ppt. Therefore, it must be emphasized that much of the data presented in this report was derived from extrapolated curves and should be considered as screening quality data only.

Sample results are summarized in Table 1. The average values reported represent samples in which there was detectable analyte. Sample values that were less than the limit of detection were not included in the average value calculations. Please refer to Section 5 for individual sample results.

Table 1. Summary of Perfluorinated Acid Results

Mean	3.33	2.84	NR[2]	0.954	4.61	1.25	NR[2]	0.623	<LOD
Median	3.33	2.5	NR[2]	0.954	4.59	1.09	NR[2]	0.660	<LOD
High	7.08	3.56	NR[2]	1.02	23.0	2.63	NR[2]	0.842	<LOD
Low	0.507	2.45	NR[2]	0.888	0.477	0.337	NR[2]	0.312	<LOD
Standard Deviation	2.17	0.627	NR[2]	0.0933	3.64	0.571	NR[2]	0.208	ND[1]
Number of Samples with Detectable Analyte	20/36	3/36	0/36	2/36	35/36	34/36	0/36	6/36	0/36

ND[1] = Not Detected. This analyte was not detected in any of the samples.
NR[2] = Not Reported. This analyte was detected in one of the 36 samples but appears to be a contaminant or an anomaly; the sample will be reanalyzed and the results will be reported in the definitive study.

Table 2. Summary of Other Perfluorinated and Highly Fluorinated Analyte Results

Mean	<LOD	0.0776	0.0348	3.27	16.3	<LOD	0.0839
Median	<LOD	0.0401	0.0339	3.27	16.9	<LOD	0.0819
High	<LOD	0.199	0.0706	5.80	32.8	<LOD	0.155
Low	<LOD	0.0311	0.0210	0.780	2.71	<LOD	0.0480
Standard Deviation	ND[1]	0.0812	0.0122	1.52	6.71	ND[1]	0.0257
Fraction of Samples with Detectable Analyte	0/36	4/36	23/36	36/36	36/36	0/36	36/36

ND[1] = Not Detected. This analyte was not detected in any of the samples.

Peaks were observed in the chromatograms corresponding to linear and branched isomers for PFOS and PFOA. Relative branched and linear isomer ratios were determined for both analytes and are presented below (the human serum value represents an average of % branched isomers for all of the human serum samples that contained detectable levels of each analyte). The % branched isomer results for individual samples are presented in Table 12. In addition, well-characterized lots of PFOS (Lot 171) and PFOA (Lot 332) were analyzed together as a matrix spike (QC050203-004) in rabbit serum. Data from that analysis was evaluated for branched and linear isomer distribution and agrees well with previously obtained NMR data.

Table 3. Summary of PFOA and PFOS Isomer Ratios

Sample	[illegible]	[illegible]
Human Serum, average	6%	39%
Matrix Spike (QC050203-004)	23%	32%
TCR-123 (99030-030) PFOA Lot 332 (NMR characterized value)	22%	
TCR-696 PFOS Lot 171 (NMR characterized value)		31%

2. Reference Substances

Table 4. Reference Substances

[illegible]	[illegible]	Source	[illegible]	[illegible]	[illegible]
Heptafluorobutyric Acid	PFBA (C4 Acid)	Aldrich	TCR-757	99.2%	0.995
Nonafluoropentanoic acid	NFPA (C5 Acid)	Aldrich	TCR-333 (00017-045)	96.5%	0.996
Perfluorohexanoic acid	PFHA (C6 Acid)	Oakwood Products	TCR-673	97.7%	0.997
Tridecafluoroheptanoic acid	TDHA (C7 Acid)	Aldrich	TCR-267	98.2%	0.997
Perfluorooctanoic acid	PFOA (C8 Acid)	3M	TCR-123 (99030-030)	95.0%	0.958
Heptadecafluorononanoic acid	C9 Acid	Oakwood Products	TCR-618	98.0%	0.998
Nonadecafluorodecanoic acid	C10 Acid	Oakwood Products	TCR-36	98.01%	0.998
Perfluoroundecanoic acid	C11 Acid	Oakwood Products	TCR-619	96.4%	0.998
Perfluorododecanoic acid	C12 Acid	Oakwood Products	TCR-37 (SD037)	99.65%	0.998
Perfluorobutanesulfonamide	FBSA	3M	TCR-314 (00017-026)	unknown	0.997
Perfluorooctanesulfonamide	FOSA	3M	TCR-280 (99131-038)	98.94%	0.997
Perfluorobutane sulfonate	PFBS	3M	TCR-282 (99131-040)	97.3%	0.885
Perfluorohexane sulfonate	PFHS	3M	TCR-83 (SE036)	98.6%	0.911
Perfluorooctane sulfonate	PFOS	3M	TCR-696	86.4%	0.927
1H, 1H, 2H, 2H, perfluorooctanesulfonic acid	THPFOS	SynQuest Labs	TCR-343 (00017-055)	93.5%	0.998
1H, 1H, 2H, 2H, perfluorodecane sulfonate	THPFDS	Pace/3M	TCR-627	94.7%	0.998
Perfluorooctanoic acid, isotopically labeled	^{13}C PFOA	Perkin Elmer	TCR-744	>97%	0.998

The samples for this study were prepared as follows:

1. The serum samples were allowed to thaw and warm to room temperature.
2. One milliliter of serum (or water in the case of method blanks) was added to a 15 mL screw-capped polypropylene centrifuge tube.
3. Matrix spikes and method blanks were prepared by spiking the serum (or water) with the analytes of interest. In addition, ^{13}C PFOA was added as a surrogate to all serum samples, calibration standards, matrix blanks, and method blanks at a concentration of 3.01 ng/mL to evaluate extraction method performance
4. Two milliliters of acetonitrile were added to the serum and the centrifuge tubes were capped and vortex mixed, allowing the serum proteins to precipitate.
5. The tubes were centrifuged at approximately 3000 rcf for 20 minutes to pellet the precipitate.
6. After removal from the centrifuge, the supernatant was transferred to an autovial for analysis.

Quantification was accomplished by high performance liquid chromatography tandem mass spectrometry. The following parameters were used:

Liquid Chromatograph: Hewlett-Packard® Series 1100 Liquid Chromatograph system
Analytical column: Keystone® Betasil™ C_{18} 2x100mm, 5µm particle size
Column temperature: 30 °C
Stop Time: 20.0 minutes
Flow rate: 300 µL/min
Injection volume: 5 µL
Mobile phase components:
Solvent A: 2.0 mM ammonium acetate in ASTM Type I water
Solvent B: HPLC Grade Methanol
Solvent Gradient:

Time	%B
0.00	20%
1.00	20%
14.50	90%
15.50	90%
16.50	20%
20.00	20%

Mass Spectrometer: MDX Sciex® API 4000 Q-Trap Mass Spectrometer Detector
Software: Analyst™ Version 1.4
Ionization Mode: Electrospray Negative
Temperature: 450 °C
GS1: 35
GS2: 45
Entrance Potential: 10
Analysis Type: Multiple Reaction Monitoring (MRM)

Table 5. Acquisition Parameters

PFBA (C_4 acid)	213.0	168.9	-30
$NFPAC_5$ Acid	262.9	219.0	-30
PFHA (C_6 acid)	313.0	268.7, 118.9	-45
TDHA (C_7 Acid)	362.9	318.7, 168.8, 118.9	-35
PFOA (C_8 Acid)	413.0	368.9, 219.0, 169.0	-40
C_9 Acid	463.0	418.7, 268.9, 218.9	-50
C_{10} Acid	512.9	468.8, 218.9, 269.1	-100
C_{11} Acid	563.0	518.7, 268.9, 218.8	-45
C_{12} Acid	613.0	568.7, 168.9, 318.7	-50
FBSA	297.9	78.0	-55
FOSA	497.9	77.9	-90
PFBS	298.9	98.9, 79.9	-65
PFHS	398.9	98.9, 80.0	-95
PFOS	498.9	80.0, 99.2, 130.0	-80
THPFOS	426.9	406.8, 80.9	-70
THPFDS	526.9	506.7, 81.0	-70
PFOA [1,2-^{13}C]	414.9	369.8	-40

When multiple transitions were monitored, the total ion current (TIC) was used to quantify each analyte, thereby providing greater sensitivity than individual Q3 ion measurements.

4.1 Calibration

4.1.1 Calibration Curve

An extracted calibration curve was formed by spiking rabbit serum at prescribed levels and extracting the curve in the same manner as the samples. Stock solutions were individually prepared from neat fluorochemicals and diluted in methanol (except PFBA, which was prepared in Milli-Q water to prevent esterification from occurring). Samples were quantified versus an 8-point extracted calibration curve. The regression analysis was performed using a quadratic curve fit weighted 1/x.

Calibration standards were within ± 20% (±25% for the Lower Limit of Quantitation (LLOQ)) with a coefficient of determination >0.995. Low-level calibration standards, which were not within ±25%, were not included in the calibration curve. Calibration standards that were not greater than twice the method blanks were not included in the calibration curve.

4.1.2 Continuing Calibration Verification (CCV)

A Continuing Calibration Verification (CCV) was run at least once every 10 samples, bracketing the samples with the calibration curve. The CCV recovery was within ±25% for all analytes.

4.1.3 System Suitability

Five system suitability standards were analyzed before the initial calibration curve and after the last CCV. These samples had area counts with a %RSD of ≤5% and a retention time %RSD of ≤2% when evaluated independently except for the following instances:

Several individual transitions monitored for the C_{10} Acid failed the retention time system suitability criteria. However, the data reported was derived from the quantitation of the TIC, which passed system suitability requirements.

All of the system suitability sets for FBSA area counts failed. The %RSDs were 6.2%, 7.5% and 8.4%. However, no samples had area counts above the LLOQ for this analyte therefore the data was accepted.

4.1.4 Lower Limit of Quantitation (LLOQ)

The LLOQ for each analyte was equal to the lowest standard in the calibration curve that met accuracy requirements of 75% -125%.

4.1.5 Limit of Detection (LOD)

The LOD for each analyte was equal to the area counts (converted to a concentration) that were 2-3 times above the method blank.

4.1.6 Blanks

Method Blanks

All of the method blanks in this study had area counts < 50% of the LLOQ.

Matrix Blanks

Two matrix blanks were prepared in rabbit serum and analyzed with the samples. One matrix blank contained endogenous levels of PFOS that were above the LLOQ. An average endogenous value for PFOS was determined by substituting a number equal to ½ the LLOQ for BLK050203-003.

Table 6. Average Endogenous PFOS Value Determination

	PFOS
LLOQ (ng/mL)	0.306
Blk050203-003	< LLOQ
Blk050203-004	0.477
Average:	0.315

The average endogenous PFOS value was used to correct the matrix spike response for endogenous levels of PFOS in rabbit serum.

4.1.7 Solvent Blanks

Acetonitrile blanks were analyzed throughout the analytical run. Analyte response was less than ½ the LLOQ in all cases.

4.1.8 Surrogate

^{13}C PFOA was added as a surrogate to all serum samples, calibration standards, matrix blanks, and method blanks at a concentration of 3.01 ng/mL to evaluate the extraction method performance. All surrogate recoveries were within ±20%.

4.2 Laboratory Control Spikes (LCS)

Six Laboratory Control Spikes (LCS) were prepared in rabbit serum and analyzed with the samples. Results were corrected for endogenous levels of analyte in the matrix (where appropriate), and are presented below.

Table 7. Matrix Spike Recoveries in Rabbit Serum

PFBA (C4 acid) LOD = 0.426 ng/mL LLOQ = 3.00 ng/mL	QC050203-001	3.00	< LLOQ	NA	NA
	QC050203-002	3.00	< LLOQ	NA	
	QC050203-003	3.00	< LLOQ	NA	
	QC050203-004	75.6	70	93%	1.0%
	QC050203-005	75.6	71.3	94%	
	QC050203-006	75.6	70.2	93%	
NFPA (C5 acid) LOD = 4.53 ng/mL LLOQ = 15.3 ng/mL	QC050203-001	3.06	< LLOQ	NA	NA
	QC050203-002	3.06	< LLOQ	NA	
	QC050203-003	3.06	< LLOQ	NA	
	QC050203-004	77.7	68.00	88%	17.9%
	QC050203-005	77.7	80.50	104%	
	QC050203-006	77.7	97.20	125%	

NA = Not Applicable

Table 7. Matrix Spike Recoveries in Rabbit Serum (continued)

PFHA (C6 acid) LLOQ = 15.1 ng/mL	QC050203-001	3.03	< LLOQ	NA	NA
	QC050203-002	3.03	< LLOQ	NA	
	QC050203-003	3.03	< LLOQ	NA	
	QC050203-004	76.8	62.4	81%	20.4%
	QC050203-005	76.8	77.2	101%	
	QC050203-006	76.8	94.2	123%	
TDHA (C7 acid) LLOQ = 3.03 ng/mL	QC050203-001	3.03	< LLOQ	NA	NA
	QC050203-002	3.03	< LLOQ	NA	
	QC050203-003	3.03	< LLOQ	NA	
	QC050203-004	76.5	66.9	87%	17.7%
	QC050203-005	76.5	80.1	105%	
	QC050203-006	76.5	95.4	125%	
C10 ACID LLOQ = 3.12 ng/mL	QC050203-001	3.12	< LLOQ	NA	NA
	QC050203-002	3.12	< LLOQ	NA	
	QC050203-003	3.12	< LLOQ	NA	
	QC050203-004	78.6	65.4	83%	12%
	QC050203-005	78.6	71.9	91%	
	QC050203-006	78.6	83.1	106%	
C11 ACID LLOQ = 0.297 ng/mL	QC050203-001	2.97	2.98	100%	5.1%
	QC050203-002	2.97	2.71	91%	
	QC050203-003	2.97	2.95	99%	
	QC050203-004	75.3	75.8	101%	1.8%
	QC050203-005	75.3	78.3	104%	
	QC050203-006	75.3	78.2	104%	
C12 ACID LLOQ = 0.747 ng/mL	QC050203-001	3.00	3.04	101%	3.8%
	QC050203-002	3.00	3.13	104%	
	QC050203-003	3.00	3.28	109%	
	QC050203-004	75.6	76.2	101%	3.9%
	QC050203-005	75.6	82.2	109%	
	QC050203-006	75.6	78.3	104%	

NA = Not Applicable

Table 7. Matrix Spike Recoveries in Rabbit Serum (continued)

^{13}C PFOA	QC050203-001	3.01	2.81	93%	4.8%
	QC050203-002	3.01	3.06	102%	
	QC050203-003	3.01	2.94	98%	
	QC050203-004	3.01	2.68	89%	
	QC050203-005	3.01	2.91	97%	
	QC050203-006	3.01	2.75	91%	
C9 Acid LLOQ = 0.303 ng/mL	QC050202-001	3.03	3.23	107%	4.7%
	QC050202-002	3.03	3.04	100%	
	QC050202-003	3.03	2.95	97%	
	QC050202-004	75.4	77.4	103%	5.3%
	QC050202-005	75.4	75.8	101%	
	QC050202-006	75.4	69.9	93%	
PFOA LLOQ = 0.298 ng/mL	QC050202-001	2.98	2.93	98%	6.8%
	QC050202-002	2.98	2.58	87%	
	QC050202-003	2.98	2.65	89%	
	QC050202-004	75.6	75.2	99%	1.6%
	QC050202-005	75.6	76.4	101%	
	QC050202-006	75.6	77.6	103%	
FBSA LLOQ = 0.303 ng/mL	QC050203-001	3.03	2.96	98%	0.7%
	QC050203-002	3.03	2.92	96%	
	QC050203-003	3.03	2.95	97%	
	QC050203-004	76.5	75.9	99%	1.4%
	QC050203-005	76.5	77.9	102%	
	QC050203-006	76.5	77.7	102%	
FOSA LLOQ = 0.300 ng/mL	QC050203-001	3.00	2.78	93%	0.8%
	QC050203-002	3.00	2.74	91%	
	QC050203-003	3.00	2.74	91%	
	QC050203-004	75.9	76.6	101%	0.7%
	QC050203-005	75.9	77.1	102%	
	QC050203-006	75.9	76.0	100%	

Table 7. Matrix Spike Recoveries in Rabbit Serum (continued)

PFBS LLOQ = 0.303 ng/mL	QC050203-001	3.03	3.06	101%	1.1%
	QC050203-002	3.03	3.00	99%	
	QC050203-003	3.03	3.01	99%	
	QC050203-004	76.8	75.8	99%	1.6%
	QC050203-005	76.8	78.3	102%	
	QC050203-006	76.8	77.2	101%	
PFHS LLOQ = 0.315 ng/mL	QC050203-001	3.15	3.04	97%	0.8%
	QC050203-002	3.15	3.08	98%	
	QC050203-003	3.15	3.04	97%	
	QC050203-004	79.8	78.2	98%	1.1%
	QC050203-005	79.8	79.9	100%	
	QC050203-006	79.8	78.6	98%	
THPFOS LLOQ = 0.780 ng/mL	QC050203-001	3.12	3.02	97%	3.8%
	QC050203-002	3.12	2.83	91%	
	QC050203-003	3.12	2.83	91%	
	QC050203-004	78.9	66.0	84%	19%
	QC050203-005	78.9	80.0	101%	
	QC050203-006	78.9	96.5	122%	
THPFDS LLOQ = 0.303 ng/mL	QC050203-001	3.03	2.96	98%	2.8%
	QC050203-002	3.03	3.00	99%	
	QC050203-003	3.03	3.12	103%	
	QC050203-004	76.5	77.9	102%	1.3%
	QC050203-005	76.5	79.6	104%	
	QC050203-006	76.5	78.2	102%	

Table 8. PFOS Matrix Spike Recovery in Rabbit Serum, Corrected for Endogenous Levels

PFOS LLOQ=0.303 ng/mL	QC050202-001	3.06	3.17	0.315	2.86	93%	2.5%
	QC050202-002	3.06	3.03	0.315	2.72	89%	
	QC050202-003	3.06	3.1	0.315	2.79	91%	
	QC050202-004	77.4	77.4	0.315	77.1	100%	1.1%
	QC050202-005	77.4	77.6	0.315	77.3	100%	
	QC050202-006	77.4	76.1	0.315	75.8	98%	

4.3 Accuracy

The accuracy for each analyte reported was calculated by taking the average of the matrix spike recoveries for that analyte. The results are presented below.

Table 9. Method Accuracy for Individual Analytes

Analyte	% Accuracy	Analyte	% Accuracy
PFBA*	± 7%	C12 Acid	± 5%
NFPA*	± 6%	FBSA	± 1%
PFHA*	± 2%	FOSA	± 4%
C7 Acid*	± 6%	PFBS	± 1%
PFOA	± 4%	PFHS	± 2%
C9 Acid	± 1%	PFOS	± 5%
C10 Acid*	± 7%	THPFOS	± 2%
C11 Acid	± 1%	THPFDS	± 1%

*The % Accuracy reported for this analyte is based on the high concentration spike recovery data only because the low concentration spike recoveries were less than the LLOQ.

4.4 Sample Related Comments

The extraction and analysis of the samples was performed utilizing a non-validated method. The matrix spike results, ^{13}C PFOA surrogate recoveries, and the extrapolated calibration curve (between the LLOQ and LOD) should be considered when evaluating the accuracy and precision of the sample results.

5 Individual Sample Data

Tables 10 and 11 summarize individual sample data.

Table 10. Perfluorinated Acid Results

TNA 6840	5.19	2.50	<LOD	<LOD	5.48	1.52	<LOD	<LOD	<LOD
TNA 6843	0.990*	<LOD	<LOD	<LOD	0.581	0.574	<LOD	<LOD	<LOD
TNA 6847	0.507*	<LOD	<LOD	<LOD	4.61	0.976	<LOD	<LOD	<LOD
TNA 6934	<LOD	<LOD	<LOD	<LOD	7.69	2.26	<LOD	<LOD	<LOD
TNA 6935	1.17*	<LOD	<LOD	<LOD	5.19	1.63	<LOD	0.451	<LOD
TNA 6944	7.08	<LOD	<LOD	<LOD	7.38	1.45	<LOD	<LOD	<LOD
TNA 6959	4.91	<LOD	<LOD	<LOD	3.13	2.45	<LOD	0.700	<LOD
TNA 6960	3.32	<LOD	<LOD	<LOD	4.59	1.11	<LOD	<LOD	<LOD
TNA 6967	4.09	<LOD	<LOD	<LOD	5.30	1.62	<LOD	<LOD	<LOD
TNA 6973	6.73	3.56	<LOD	0.888*	5.51	1.64	<LOD	<LOD	<LOD
TNA 6975	2.95*	<LOD	<LOD	<LOD	5.52	1.31	<LOD	<LOD	<LOD

Table 10. Perfluorinated Acid Results (continued)

[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	0.298	0.303	[illegible]	[illegible]	0.732
TNA 6977**	5.04	<LOD	<LOD	<LOD	0.477	<LOD	<LOD	<LOD	<LOD
TNA 6981	6.76	2.45	<LOD	<LOD	4.12	1.28	<LOD	<LOD	<LOD
TNA 6992	0.690*	<LOD	<LOD	<LOD	4.76	0.912	<LOD	<LOD	<LOD
TNA 6994	<LOD	<LOD	<LOD	<LOD	1.15	0.337	<LOD	<LOD	<LOD
TNA 6995	1.98*	<LOD	18.4***	1.02*	3.58	2.63	<LOD	0.815	<LOD
TNA 6997	3.90	<LOD	<LOD	<LOD	3.46	2.52	<LOD	0.842	<LOD
TNA 7000	3.34	<LOD	<LOD	<LOD	23.0	1.08	<LOD	<LOD	<LOD
TNA 7006	3.19	<LOD	<LOD	<LOD	4.18	1.09	<LOD	<LOD	<LOD
TNA 6938	<LOD	<LOD	<LOD	<LOD	5.00	0.979	<LOD	<LOD	<LOD
TNA 6939	<LOD	<LOD	<LOD	<LOD	2.32	0.941	<LOD	0.312	<LOD
TNA 6941	3.56	<LOD	<LOD	<LOD	5.82	1.69	<LOD	0.620	<LOD
TNA 6950	<LOD	<LOD	<LOD	<LOD	3.87	0.723	14.0***	<LOD	<LOD
TNA 6953	0.621*	<LOD	<LOD	<LOD	5.49	0.725	<LOD	<LOD	<LOD
TNA 6955	<LOD	<LOD	<LOD	<LOD	2.54	0.562	<LOD	<LOD	<LOD
TNA 6962	<LOD	<LOD	<LOD	<LOD	4.68	0.941	<LOD	<LOD	<LOD
TNA 6964	<LOD	<LOD	<LOD	<LOD	5.61	1.58	<LOD	<LOD	<LOD
TNA 6966	<LOD	<LOD	<LOD	<LOD	4.73	1.35	<LOD	<LOD	<LOD
TNA 6970	<LOD	<LOD	<LOD	<LOD	6.00	1.42	<LOD	<LOD	<LOD
TNA 6972	<LOD	<LOD	<LOD	<LOD	3.40	1.09	<LOD	<LOD	<LOD
TNA 6978**	<LOD	<LOD	<LOD	<LOD	< LOD	< LOD	<LOD	<LOD	<LOD
TNA 6979	<LOD	<LOD	<LOD	<LOD	3.32	0.981	<LOD	<LOD	<LOD
TNA 6982	<LOD	<LOD	<LOD	<LOD	1.48	0.664	<LOD	<LOD	<LOD
TNA 6984	<LOD	<LOD	<LOD	<LOD	2.84	0.955	<LOD	<LOD	<LOD
TNA 6986	0.530*	<LOD	<LOD	<LOD	2.08	0.645	<LOD	<LOD	<LOD
TNA 6988	<LOD	<LOD	<LOD	<LOD	2.57	0.881	<LOD	<LOD	<LOD
Mean	3.33	2.84	NA	0.954*	4.61	1.25	NA	0.623	NA
Median	3.33	2.50	NA	0.954*	4.59	1.09	NA	0.660	NA
High	7.08	3.56	NA	1.02*	23.0	2.63	NA	0.842	NA
Low	0.507*	2.45	NA	0.888*	0.477	0.337	NA	0.312	NA
Standard Deviation	2.17	0.627	NA	0.0933	3.64	0.571	NA	0.208	NA
# OF HITS	20	3	0	2	35	34	0	6	0

NA = Not Applicable
*Analyte concentration was below the LLOQ and above the LOD.
**TN-A-6977 and TN-A-6978 are from the same lot of pooled serum.
***Analyte was detected in one of the 36 samples but appears to be a contaminant or an anomaly; the sample will be reanalyzed and the results will be reported in the definitive study.
Based on ^{13}C PFOA surrogate recovery, the overall method uncertainty is +/- 20%.

Table 11. Sample Results for Additional Perfluorinated and Highly Fluorinated Analytes

TNA 6840	<LOD	<LOD	<LOD	4.39	23.0	<LOD	0.124*
TNA 6843	<LOD	<LOD	0.0228*	0.780	3.42	<LOD	0.155*
TNA 6847	<LOD	0.0458*	<LOD	2.05	23.7	<LOD	0.145*
TNA 6934	<LOD	<LOD	0.0304*	2.41	32.8	<LOD	0.0531*
TNA 6935	<LOD	<LOD	0.0433*	2.14	16.2	<LOD	0.0820*
TNA 6944	<LOD	<LOD	0.0706*	4.73	20.4	<LOD	0.0860*
TNA 6959	<LOD	<LOD	<LOD	3.75	14.8	<LOD	0.0649*
TNA 6960	<LOD	<LOD	0.0282*	4.52	19.6	<LOD	0.0819*
TNA 6967	<LOD	0.199*	0.0358*	4.27	21.3	<LOD	0.0857*
TNA 6973	<LOD	<LOD	<LOD	5.18	20.5	<LOD	0.0818*
TNA 6975	<LOD	<LOD	0.0393*	4.96	19.4	<LOD	0.0822*
TNA 6977**	<LOD	<LOD	0.0339*	0.844	3.01	<LOD	0.0916*
TNA 6981	<LOD	0.0311*	0.0313*	1.77	17.4	<LOD	0.103*
TNA 6992	<LOD	<LOD	0.0429*	4.45	19.8	<LOD	0.0826*
TNA 6994	<LOD	<LOD	<LOD	1.71	3.50	<LOD	0.0616*
TNA 6995	<LOD	<LOD	0.0366*	3.79	15.1	<LOD	0.0853*
TNA 6997	<LOD	<LOD	0.0479*	3.81	15.2	<LOD	0.0707*
TNA 7000	<LOD	<LOD	0.0568*	5.68	22.9	<LOD	0.0638*
TNA 7008	<LOD	<LOD	0.0412*	4.90	20.2	<LOD	0.0667*
TNA 6938	<LOD	<LOD	0.0226*	2.78	15.6	<LOD	0.0601*
TNA 6939	<LOD	<LOD	<LOD	1.79	6.23	<LOD	0.0625*
TNA 6941	<LOD	<LOD	0.0224*	3.06	23.4	<LOD	0.0577*
TNA 6950	<LOD	<LOD	<LOD	3.24	14.3	<LOD	0.0909*
TNA 6953	<LOD	<LOD	0.0226*	5.24	22.1	<LOD	0.0480*
TNA 6955	<LOD	<LOD	<LOD	3.29	10.1	<LOD	0.0493*
TNA 6962	<LOD	<LOD	0.0312*	5.22	19.8	<LOD	0.0705*
TNA 6964	<LOD	<LOD	0.0374*	5.80	21.7	<LOD	0.101*
TNA 6966	<LOD	<LOD	0.0229*	4.62	17.5	<LOD	0.0620*
TNA 6970	<LOD	<LOD	<LOD	4.06	17.0	<LOD	0.137*
TNA 6972	<LOD	<LOD	0.0345*	3.09	12.7	<LOD	0.0653*
TNA 6978**	<LOD	<LOD	0.0210*	0.945	2.71	<LOD	0.0998*
TNA 6979	<LOD	0.0344*	0.0228*	2.31	16.7	<LOD	0.102*
TNA 6982	<LOD	<LOD	<LOD	1.26	9.37	<LOD	0.0790*
TNA 6984	<LOD	<LOD	<LOD	1.73	15.8	<LOD	0.0911*
TNA 6986	<LOD	<LOD	<LOD	1.41	11.3	<LOD	0.0683*

Table 11. Sample Results for Additional Perfluorinated and Highly Fluorinated Analytes (continued)

TNA 6988	<LOD	<LOD	<LOD	1.79	16.5	<LOD	0.0979*
Mean	NA	0.0776*	0.0348*	3.27	16.3	NA	0.0839*
Median	NA	0.0401*	0.0339*	3.27	16.9	NA	0.0819*
High	NA	0.199*	0.0706*	5.80	32.8	NA	0.155*
Low	NA	0.0311*	0.0210*	0.780	2.71	NA	0.048*
Standard Deviation	NA	0.0812	0.0122	1.52	6.71	NA	0.0257
# OF HITS	0	4	23	36	36	0	36

NA = Not Applicable
*Analyte concentration was below the LLOQ and above the LOD.
**TN-A-6977 and –6978 are from the same lot of pooled serum.
Based on ^{13}C PFOA surrogate recovery, the overall method uncertainty is +/- 20%.

Table 12. Isomer Ratios for Individual Samples

TNA 6840	California	11%	40%
TNA 6843	Missouri	13%	40%
TNA 6847	Pennsylvania	4.1%	43%
TNA 6934	Pennsylvania	0.8%	45%
TNA 6935	New York	12%	37%
TNA 6944	Michigan	3.4%	35%
TNA 6959	Virginia	4.3%	40%
TNA 6960	Virginia	4.5%	38%
TNA 6967	Maryland	9.5%	43%
TNA 6973	Montreal, Quebec	4.3%	42%
TNA 6975	Montreal, Quebec	4.0%	42%
TNA 6977**	Massachusetts	< LLOQ*	39%
TNA 6981	Texas	3.7%	43%
TNA 6992	California	4.2%	40%
TNA 6994	California	4.0%	39%
TNA 6995	California	6.0%	41%
TNA 6997	California	4.0%	41%
TNA 7000	California	17%	41%
TNA 7006	California	6.3%	41%
TNA 6938	New York	2.7%	44%
TNA 6939	New York	4.0%	35%
TNA 6941	New York	3.0%	36%

Table 12. Isomer Ratios for Individual Samples (continued)

TNA 6950	Michigan	0.3%	39%
TNA 6953	Michigan	10%	39%
TNA 6955	Michigan	3.0%	35%
TNA 6962	Virginia	6.8%	41%
TNA 6964	Virginia	5.4%	42%
TNA 6966	Virginia	7.9%	40%
TNA 6970	Maryland	6.7%	39%
TNA 6972	Maryland	5.1%	38%
TNA 6978**	Massachusetts	< LLOQ*	41%
TNA 6979	Massachusetts	7.6%	30%
TNA 6982	Texas	6.0%	41%
TNA 6984	Texas	1.3%	31%
TNA 6986	Texas	0.1%	43%
TNA 6988	Texas	4.5%	33%
	Average:	**6%**	**39%**
	Standard Deviation	**3%**	**3%**

*Sample value was < LLOQ
**TN-A-6977 and TN-A-6978 are from the same lot of pooled serum.

All original raw data and the analytical report have been archived at the 3M Environmental Laboratory. The test materials and analytical reference standard reserve samples, as well as the samples pertaining to this project are archived at the 3M Environmental Laboratory.

Reserve samples, digital copies of original data and all original paper data will be retained in the archives of 3M Environmental Laboratory for a period of at least 10 years following report signing.

Under the conditions of the study, endogenous fluorochemicals were found to be present in all samples. PFHS, PFOS, and THPFDS were detected in every lot of serum while PFOA was present in detectable levels in all but one sample. Conversely, FBSA, THPFOS, and the C_{12} acid were not detected in any of the samples using the present methodology. ^{13}C PFOA surrogate recoveries were all within ±20%.

[redacted]

Mark Ellefson, Project Lead — Date: 08/12/05

William Reagen, Laboratory Manager — Date: 08/12/05

The 3M Environmental Laboratory's Quality Assurance Unit has audited the data and report for this project.

Quality Assurance Representative — Date

8. Signatures

Mark Ellefson, Project Lead — Date: 08/12/05

William Reagen, Laboratory Manager — Date: 08/12/05

The 3M Environmental Laboratory's Quality Assurance Unit has audited the data and report for this project.

Quality Assurance Representative — Date: 8/12/05